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[54] [Title of Invention]

Method of leak detection of selective permeable membrane module

[57] [Summary]
[Issue] To provide a method of leak detection that permits the identification and repair of leakage sites in a reverse osmotic membrane and nano-filtration membrane module without contamination or damage to permeability.
[Means of Resolution] Fluorescent dye solution having molecular weight of 300 to 3000 is added to a feed liquid, and any dye leaking from defects in the membrane module is detected via fluorescence.

**[Scope of Patent Claim]**

[Claim 1] A method of leak detection in a selective permeable membrane module in which fluorescent dye solution having molecular weight of 300 to 3000 is used to detect any fluorescence leaking from defects in the membrane module to identify the leak.
[Claim 2] The method of Claim 1 in which the selective permeable membrane module is a hollow fiber selective permeable membrane module.
[Claim 3] The method of Claims 1 or 2 in which the fluorescent dye is a fluorescent water-soluble food dye.
[Claim 4] The method of Claims 1 or 2 in which the selective permeable membrane module comprises cellulose acetate or cellulose triacetate, and the fluorescent dye is one type or a mixture of two or more types of diamino stilbene disulfonate derivative.
[Claim 5] The method of Claims 1 or 2 in which the active separation layer of the selective permeable membrane is a polymer whose main constituents are piperazine and trimesic acid residue, and in which the fluorescent dye is food red No.104 and/or food red No.106.
[Claim 6] The method of Claims 1 or 2 in which the active separation layer of the selective permeable membrane is a polymer whose main constituents are piperazine and trimesic acid residue, and in which the fluorescent dye is eosin and/or derivatives thereof.
**[Detailed Description of the Invention]**
[0001]
**[Field of Industrial Utilization]** The present invention concerns a method of detecting defects in selective permeable membrane modules used in water purification and in cleanup of waste water, especially a method of detecting defects in membrane modules used in separation, purification, recovery when pressure is used as the driving power, and especially a method of detecting defects by identifying leakage sites in reverse osmotic membranes and nano-filtration membrane modules.
[0002] Reverse osmotic membranes are used in desalination of sea water and in ultrapure water. Their application in advanced processing of tap water and in advanced water purifiers has been examined in recent years. Extremely low operating pressure is employed in these applications, and nano-filtration membranes having low desalination rates have been developed. Defects often develop in membrane modules, including defects created during membrane production, damage caused by handling, or cracks forming at the junction of a membrane and container. On the other hand, even small defects that form on a membrane surface reduce the separation performance, permit leakage of viruses or endotoxins, and damage the integrity of the membrane module. The repair of such defects is linked to the production of inexpensive membrane modules having integrity. Identification of leakage sites is essential to the repair of membrane module having defects.
[0003]
**[Related Art]** The two principal methods of leak detection are the method utilizing air leaking from defects and the method utilizing anomalous liquid permeability. Methods utilizing air include the methods presented in Japanese Kokai Publication Sho-62-140607 and Japanese Kokai Publication Sho-63-84606 in which air issued in water is observed; the method presented in Japanese Kokai Publication Hei-5-137977 in which the pressure changes corresponding to the generation of air bubbles; the method presented in Japanese Kokai Publication Sho-62-144705 in which maintenance of the degree of vacuum is measured; and the method presented in Japanese Kokai Publication Hei-1-307409 in which the residual air pressure is measured to determine the presence of leaks. The method of measuring the amount of permeation from airflow resistance, as presented in Japanese Kokai Publication Sho-61-249507, can also be utilized in leak detection. In Japanese Kokai Publication Sho-61-220710 and Japanese Kokai Publication Hei-3-127614, the existence of leaks is detected by measuring the number of fine particles in a permeating stream of gas containing a suspension of fine particles using a particle counter. Other detection methods that have been devised employ tactile sensation of a stream in an air discharge, various types of measurement instruments of differences in the refractive index, differences in the density or differences in the temperature; or apply visualization technology. Methods utilizing anomalous liquid permeability have also been devised. Membrane leakage is detected in the method presented in Japanese Kokai Publication Sho-62-213811 by detecting the quality of filtrate; in the method presented in Japanese Kokai Publication Sho-59-183807 by detecting fluctuations in the values of liquid on the secondary side; in the method presented in Japanese Kokai Publication Sho-60-25510 by detecting pressure fluctuations in feed liquid. Leakage is detected in the method presented in Japanese Kokai Publication Hei-6-182164 through utilization of increase in the

water-transit pressure when filtering solids found in permeating water through a second stage close-tolerance filtration membrane; in the method presented in Japanese Kokai Publication Hei-7-248290 through utilization of a leak detection monitor such as a pressure gauge or a leak detection filter after diverting part of the permeating water; in the method presented in Japanese Kokai Publication Hei-5-157654 through utilization of the pressure elevation in a filtration chamber when gas pressurization is applied to a sealed liquid. Methods of adding solute or suspended matter to feed liquid have also been devised. For example, the method presented in Japanese Kokai Publication Hei-7-243959 detects leaks of suspended magnetic or conductive matter; the method presented in Japanese Kokai Publication Sho-59-136631 detects leaks by measuring the filtration performance or properties of permeating solution of fluorescent labeling polymer (FITC-Dxt-T-200), the method presented in Japanese Kokai Publication Hei-6-254358 is applied to permeating solution of food dye; the method presented in Japanese Kokai Publication Hei-7-132215 is applied to permeating solution of viral substitute particles. Examples of other methods include the method presented in Japanese Kokai Publication Hei-3-174225 in which defects in a hollow fiber inclusion are determined based on the intensity of scattering of permeating light in the inclusion; the method presented in Japanese Kokai Publication Hei-3-174226 in which defects are determined based on the intensity of reflected light; and the method presented in Japanese Kokai Publication Hei-3-213129 in which defects are determined based on changes in the polarization of permeating light. A coating layer to which dye is added is created in the method presented in Japanese Kokai Publication Sho-62-136225, while hydrophobic or hydrophilic paint is applied to a vapor permeation membrane in the method presented in Japanese Kokai Publication Hei-7-155567 to identify membrane defects. The method of measuring the potential difference on both sides of a membrane in the method presented in Japanese Kokai Publication Hei-6-170186 and the method of measuring the viral removal rate in the method presented in Japanese Kokai Publication Hei-5-502164 can also be utilized in defect detection.

[0004]

**[Problems Solved by the Invention]** However, methods using gases are basically experimental methods applied to close-tolerance filtration membranes. High pressure is required to generate bubbles when methods applying the generation of bubbles are used in leak detection of reverse osmotic membranes and nano-filtration membranes. This cannot be applied to leak detection of reverse osmotic membranes and nano-filtration membranes since the membranes are prone to deformation or damage when high pressure is applied. Other methods including those using gases cannot be applied since they basically do not permit identification of the leakage sites. Methods permitting visualization of gas passage (leaks) do not readily permit detection of pinholes since the equipment is so large. In addition, permeation through membrane modules suffers since their use expands the area that must be repaired. Methods using solutions or suspensions suffer attendant contamination of the membrane module when the membrane is blocked, while detection is impossible without using high concentrations when various types of solutes are used. Even if a leakage site could be identified, the permeability of a membrane module would be impaired since the membrane module is contaminated. In the method using polymer solutions, the polymer is larger than the pore diameter of the target membrane, which facilitates membrane contamination and complicates cleaning following leak detection. Minute leaks tend to be overlooked with increase in the frequency of damage to membrane performance. Solutions of dyes or pigments react with the membrane, and the membrane- or module constituents are often contaminated since a comparatively high concentration of dye is required for leak detection. As indicated above, leakage site must be identified in order to repair membrane modules, but an appropriate method has not been found to date.

[0005]

**[Means of Solving the Problems]** The inventors have discovered the following novel method of leak detection as a result of thorough research. Specifically, the present invention concerns a method of leak detection in a selective permeable membrane module in which fluorescent dye solution having molecular weight of 300 to 3000 is used to detect any fluorescence leaking from defects in the membrane module to identify the leak. Furthermore, it concerns a method of leak detection of selective permeable membrane modules in which food dye leaking from defects in a membrane module is detected via fluorescence as a result of using fluorescent, water-soluble food dyes to identify leakage sites.

[0006] The present invention is explained in detail below. Any selective permeable membrane module may be used so long as it is a membrane module constructed such that a separated membrane permitting separation and concentration of material is the main separated constituent. Reverse osmotic membranes and nano-filtration membranes are preferred. Any method of membrane production may be used so long as it produces a compound membrane comprising a uniform membrane and an asymmetrical membrane, permissible methods including casting, coating, surface reforming, interfacial polymerization, wet spinning, semi-wet spinning, or dry spinning. There is no specific limitation on the material

comprising the uniform membrane, asymmetrical membrane, or the backing member of the compound membrane. Permissible examples include polymers such as various types of polyolefins, cellulose acetate, cellulose triacetate, polyamide, polyimide, polyacrylonitrile, polysulfone, polyfluoroethylene, polyether sulphone, or derivatives thereof as well as copolymers of these. There is no specific limitation to the membrane materials comprising the separating active layer of a compound membrane. Permissible examples include polymers such as cellulose acetate, cellulose triacetate, polyamide, polyimide, polyacrylonitrile, polysulfone, polyether sulphone, derivatives thereof and copolymers with these. Desirable membrane constituents include various types of polyamides, cellulose triacetate, or cellulose acetate. Selective permeable membrane modules would be completed by processing these membranes into the required shape, housing them in suitably shaped containers, and affixing them with adhesive, etc. The membrane module may adopt any of a variety of shapes, including a spiral winding shape using flat membranes, multi-layered laminates, hollow fiber shapes, or tubular membrane shapes, but hollow fiber membrane modules would be the most effective of these in leak detection.

[0007] Fluorescent dyes in the present invention connote fluorescent material using any solvent in which they dissolve. There is no restriction on the fluorescent dye so long as it satisfies these conditions. Specific examples include all fluorescent dyes having C.I.Fluorescent Brightener numbers represented by the dye classification color index. Preferable examples include numbers 20, 24, 30, 32, 46, 48, 54, 71, 84, 85, 86, 87, 152, 156, 166, 225 226, 260, 351, 19, 21, 22, 23, 31, 33, 35, 36, 37, 40, 72, 90, 135. In addition, fluorescent material represented by eosin, rhodamine B or derivatives thereof would correspond to this general dye classification. Additional dyes include fluorescent food dyes and natural dyes, such as food red No.3, food red No.104, food red No.106, porphyrin derivatives including sodium iron chlorophyllin and sodium potassium chlorophyllin, $\beta$-carotene, norbixin, bioflamin [phonetic] derivatives, and vitamin B12 derivatives. Any of these fluorescent dyes could be used.

[0008] Desirable examples of these include aforementioned food dyes and C.I.Fluorescent Brightener 24, C.I.Fluorescent Brightener 86, C.I.Fluorescent Brightener 90, C.I.Fluorescent Brightener 260, eosin and derivatives thereof. The most desirable of these would be food red No.104, food red No.106, as well as 4,4'-diamino stilbene-2,2'-disulfonate derivative fluorescent dyes of C.I.Fluorescent Brightener 24, C.I.Fluorescent Brightener 86, C.I.Fluorescent Brightener 260, eosin and derivatives thereof.

[0009] The capacity of a membrane to obstruct fluorescent dyes is dependent on the molecular size of the fluorescent dye used. When the molecular size is small, leaks would not be detected in terms of permeation of the overall aperture of a membrane module since fluorescent dye permeates a membrane, while if the molecular size is too large, the amount of leakage from leakage sites would decrease and leaks would not be detected. Minute defects would be undetectable. When considering the capacity of a membrane to obstruct fluorescent dyes, a range of molecular weights of fluorescent dyes from 300 to 3000 would be effective. A range of 450 to 1800 would be preferable, and a range of 550 to 1200 would be most preferable.

[0010] These fluorescent dyes can be easily visualized under ultraviolet irradiation. Specifically, desired sites may be irradiated with ultraviolet light generated by an ultraviolet lamp. The use of an extremely slight amount of fluorescent dye would be sufficient since the fluorescence generated upon ultraviolet irradiation is very bright. Any method of identifying leakage sites may be used. They may be identified visually by viewing fluorescent dyes in an effluent stream, or leakage sites may be determined by capturing the fluorescence with a television camera, followed by image processing. A simple method would be to apply the method presented in Japanese Kokai Publication Hei-6-254358. This is a method in which a detection medium is attached to the discharge port of water permeating a membrane module, after which fluorescent dye discharged from defects stains the detection medium. The fluorescence generated at the spot on the medium stained by fluorescent dye is then detected. Consequently, the position corresponding to the leakage site can be detected as a fluorescent stained spot. Compared to fluorescent detection, detection via staining provides detection capability 250 times greater. For example, it permits identification of a leakage site even at 20 $\mu$g/liter. The affinity with fluorescent dye can be raised by selecting a detection medium. Leakage site can be easily identified and even minute defects such as pinholes or peeling from the membrane surface can be detected.

[0011] Concretely, a fluorescent dye solution comprising one or a mixture of two or more diamino stilbene disulfonate derivatives would be used in leak detection of selective permeable membrane modules comprising cellulose acetate or cellulose triacetate. This is a method of leak detection in which dye leaking from defects in the membrane module is detected in identification of the leakage site. Furthermore, a solution of food red No.104 or food red No.106 would be used as the fluorescent dye in leak detection of a selective permeable membrane module in which the main constituent of the active separation layer of the selective permeable membrane is a polymer comprising piperadine and trimesic acid residue. Another method of leak detection is one in which a dye solution comprising eosin and/or derivatives thereof

is used in detection of dye leaking from defects in a membrane module via the fluorescence to thereby identify leakage sites.

[0012] The method of repairing defects following identification of a leakage site varies with the type of membrane module. There is no fixed method. Rather, any of a variety of known methods may be applied. For example, an adhesive or a sealant may be applied to a leak in any shape of membrane module. An impermeable sheet may be applied to a leaks while a sealant may be applied after processing, such as shaving, in order to improve the adhesion to a leakage site in a hollow fiber membrane. Similarly, apertures in impermeable material may be sealed in the case of a hollow fiber or tube, among many available methods.

[0013]

[Working Examples] The present invention is explained below with reference to the following working examples.

[0014] (Working Example 1)

Method I of evaluating performance of membrane module

An evaluation system in which permeating water and concentrated water are circulated back to a raw water tank was used in the membrane module evaluation shown in Figure 1. A thermostat is attached to maintain a constant temperature the temperature is controlled at a specified level. Aqueous brine solution, 1500 mg/liter, is fed at 25°C, 30 kg/cm$^2$, and a valve is adjusted so that the recovery rate RC would be 75%. The amount of permeating water as well as the water quality would be measured two hours after pressurization as a rule. The electrical conductivity is measured when measuring the water quality, and it is determined in terms of the brine concentration. The water flow rate FR through the membrane module and the dechlorination rate RJ are defined by the following formulas.

(formula 1) $RC = Qp/Qf \times 100$ (%)
(formula 2) $FR = Qp \times 1.44$ (m$^3$/day)
(formula 3) $RJ = (Cf-Cp)/Cf \times 100$ (%)

Here, Qf (=Qp + Qb) represents the amount feedwater to the membrane module (liter/minute), Qp represents the water flow rate (liter/minute), Qb represents the amount of concentrated water (liter/minute). Cf represents the salt concentration in the feedwater (mg/liter) while Cp represents the salt concentration in the flowing water (mg/liter).

[0015] Method II of evaluating performance of membrane module

Aforementioned evaluation method was repeated except for altering the operating conditions to 500 mg/liter aqueous solution of magnesium sulfate, 25°C, 5 kg/cm$^2$, recovery rate RC of 50%.

[0016] Test of Escherichia coli removal

The equipment and operating conditions were in accordance with the method of evaluating the performance of the membrane module. Time required to stabilize operations: Escherichia coli culture liquid that had been precultured and stored after the elapse of two hours was added. In addition, an equal amount was added after the elapse of 20 hours. The flowing water and feedwater were extracted after the elapse of 2 more hours. Escherichia coli, K-12 strain (IFO3206) was cultured for two or more days at 36°C in BHI liquid culture medium as the Escherichia coli culture. 20 milliliters were added twice at aforementioned times per 300 liters of raw water.

[0017] Measurement of Escherichia coli number

The extracted sample was measured immediately if possible, otherwise within 24 hours following refrigeration at 5°C or less. The measurement complied with the Escherichia coli count in the trial method for factory waste water in JIS K0102-1993.

[0018] A cellulose triacetate hollow fiber membrane module (brand name HOLLOWSEP HA5330, Toyobo Co., Ltd.) was produced as a reverse osmotic membrane. A membrane whose characteristics were outside of specifications was selected and used in leak detection. The results of an Escherichia coli removal test in evaluation method I of the membrane module revealed the permeation performance through the membrane module to be 1200/cm$^3$ in flowing water versus 3,000,000/cm$^3$ in raw water at FR 28.4 m$^3$/day, RJ90, 2%. In a 500 liter raw water tank was dissolved 20 cm$^3$ of an aqueous solution of 1 g/liter of fluorescent dye C.I.Fluorescent Brightener 24 (fluorescent dye concentration of 0.04 mg/liter). Qualitative filter paper was inserted in the aperture of the membrane module as a leak detection medium, and this was operated for one hour at 10 kg/cm$^2$, 20 to 25°C. After the operation was completed, the filter paper was removed and irradiated under a fluorescent lamp to determine the leakage sites through development of fluorescence. Fluorescent stained spots, specifically, the number of leakage sites, was confirmed to be 43. The apertures of the membrane module corresponding to the leakage sites were drilled open and sealed with epoxy sealant. The membrane module was

reevaluated after the sealant cured. The results of an Escherichia coli removal test of the membrane module revealed the permeation performance through the membrane module to be 0/$cm^3$ in flowing water versus 3,000,000/$cm^3$ in raw water at FR 24.8 $m^3$/day, RJ93, 1%. The membrane module was subsequently disassembled and examined, but neither contamination nor sites of membrane staining were found.

[0019] (Working Example 2) The procedures of Working Example 1 were repeated except for the use of C.I.Fluorescent Brightener 86 as the fluorescent dye. The results are presented in Table 1.

[0020] (Working Example 3) The procedures of Working Example 1 were repeated except for the use of C.I.Fluorescent Brightener 260 as the fluorescent dye. The results are presented in Table 1.

[0021] (Comparative Example 1) The procedures of Working Example 1 were repeated except for the use of 5 mg/liter of food red No.2 as the fluorescent dye, ion exchange filter paper DEAE (manufactured by Toyo Filter Paper) as the detection medium, and observation of stained spots without fluorescence. The results are presented in Table 1. Neither significant decrease in the water flow rate FR nor obvious dark staining of hollow fiber membranes were seen upon disassembly.

[0022] (Comparative Example 2) The procedures of Working Example 1 were repeated except for the use of fluorescent labelling polymer (FITC-Dxt-T-200) synthesized by the method presented in Japanese Kokai Publication Sho-59-136631 as fluorescent dye and ion exchange filter paper CM (manufactured by Toyo Filter Paper) as the detection medium. The results are presented in Table 1. Since stained spots were not detected well, the dextran concentration was raised to 200 mg/liter and the water flow rate FR decreased significantly.

[0023] (Working Example 4) The procedures of Working Example 1 were repeated except for the use of the membrane module used in Comparative Example 2 and an aqueous solution of 0.04 mg/liter of C.I.Fluorescent Brightener 86 as the fluorescent dye. A total of 13 minute fluorescent stained spots remained, indicating that the detection capability in Comparative Example 2 was inadequate. Repairs were repeated. The results are presented in Table 1.

[0024]

[Table 1]

Leak repair results of cellulose triacetate membrane module

| Example | Name of dye (F: abbreviation for C.I.Fluorescent Brightener). | Evaluation results after repair | | |
|---|---|---|---|---|
| | | FR ($m^3$/day) | RJ (%) | Escherichia coli count in flowing liquid (number/$cm^3$) |
| Working Example 2 | F86 | 24.1 | 92.5 | 0 |
| Working Example 3 | F260 | 23.4 | 96.2 | 0 |
| Comparative Example 1 | Food red No.2 | 8.6 | 96.8 | 0 |
| Comparative Example 2 | FITC-Dxt-T-200 | 18.6 | 96.6 | 83 |
| Working Example 4 | F86 | 18.8 | 96.8 | 0 |

[0025] (Working Example 5) A composite hollow fiber selective permeable membrane module was constructed having 6000 hollow fibers in an effective length of 185 mm. A composite hollow fiber selective permeable membrane module was manufactured by interfacial polymerization using piperazine and triethylene diamine (molar ratio 99:1) as the amine constituent on polysulfone hollow fiber porous membrane (minor diameter 200 $\mu m$, major diameter 300 $\mu m$) as the nano-filtration membrane and trimesic acid chloride as the acid constituent. The membrane module was evaluated using evaluation method II. A membrane module with worse permeation performance (anticipated performance FR: 250 liters/day, RJ: 70% versus FR: no less than 270 liters/day, RJ: not more than 55%) was selected. The permeation

performance preceding leak detection was FR: 285 liters/day, RJ: 44.0%. Material having an Escherichia coli count of 2800/$cm^3$ in flowing water and 3,000,000/$cm^3$ or more in raw water was chosen. Food red No.106 was used as the dye and ion exchange filter paper DEAE (manufactured by Toyo Filter Paper) was used as the detection medium. Other than these differences, the procedures of Working Example 1 were repeated. The results are presented in Table 2.

[0026] (Working Example 6) The procedures of Working Example 5 were repeated except for the use of food red No. 104 as the dye. The results are presented in Table 2.

[0027] (Comparative Example 3) The procedures of Working Example 5 were repeated except for the use of food red No. 3 as the dye. The results are presented in Table 2. The detection capacity was insufficient using food red No. 3, and the membrane module was contaminated.

[0028] (Working Example 7) The procedures of Working Example 5 were repeated except for the use of eosin as the dye. The results are presented in Table 2.

[0029]

[Table 2]

Leak repair results of polyamide composite selective permeable membrane module

| Parameters \ example | Working Example 5 | Working Example 6 | Comparative Example 3 | Working Example 7 |
|---|---|---|---|---|
| Name of dye Concentration used (mg/liter) | Food red No. 106 0.1. | Food red No. 104 0.02. | Food red No. 3 0.2. | Eosin 0.05. |
| Leak detection medium | DEAE | DEAE | DEAE | DEAE |
| Evaluation before leak detection | | | | |
| PR (liter/day) | 285 | 305 | 270 | 275 |
| RJ (%) | 44.0 | 38.8 | 54.8 | 51.6 |
| Escherichia coli count raw water (no./$cm^3$) flowing water . | >3,000,000 2800. | >3,000,000 4500. | >3,000,000 1200. | >3,000,000 1100. |
| Leak repair count (no.) | 33 | 46 | 21 | 15 |
| Evaluation after leak repair | | | | |
| FR (liter/day) | 245 | 210 | 255 | 235 |
| RJ (%) | 66.0 | 71.2 | 59.8 | 65.5 |
| Escherichia coli count raw water | >3,000,000 0 | >3,000,000 0 | >3,000,000 94 | >3,000,000 0 |
| Contamination state | Hollow fibers slightly stained | No contamination | Red staining, massive contamination | No contamination |

[0030]

**[Action]** Detection is simplified by the fluorescence in the dye and that also permits the use of a low dye concentration. Contamination of the membrane and constituents of the selective permeable membrane module under examination are greatly reduced to virtually nil.

[0031]

**[Effects of Invention]** Consequently, the decline in water flow through a selective permeable membrane module in leak detection can be eliminated, the appearance of the selective permeable membrane module can be maintained, residue of contaminating dye can be eliminated when using this selective permeable membrane module, and a hygienic, safe water flow can be realized. A membrane module having high water permeability without defects can be manufactured inexpensively since the membrane module can be repaired without damaging the permeability.

**[Brief Description of Drawings]**

[Figure 1] A flow sheet showing the evaluation device pursuant to the present invention.

[Explanation of Notation]

1 Membrane module
2 Feedwater pipe
3 Flowing water pipe
4 Concentrated water pipe
11 Raw water tank
12 Pump
13 Valves

Figure 1